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A MANUAL  
OF  
QUANTITATIVE  
CHEMICAL ANALYSIS.

BY

E. F. LADD, B.S.,

*Professor of Chemistry in the North Dakota Agricultural College.  
and Chemist to the Government Experiment Station,  
Fargo, N. D.*

*FIRST EDITION.*

FIRST THOUSAND.

NEW YORK :  
JOHN WILEY & SONS  
LONDON : CHAPMAN & HALL, LIMITED.

1898.



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## PREFACE.

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THIS little manual is intended for the use of beginners in quantitative analysis, and is in no way designed for the use of the professional chemist.

The methods have been selected to advance the student from the simple analysis to the more complex and difficult, and when he has completed the course as laid down here he will be in a position to intelligently use and interpret the advanced works of Fresenius, Crookes, and the *Encyclopædias*. The methods selected are not the ones that would always give the most reliable results in professional work, but rather are those that represent principles that will aid in developing the reasoning power of the student; and for that reason explanations of details and reactions have been largely omitted, and the series of questions at the end of each method will test the student's knowledge of chemistry, and make it necessary that he consult other works to acquaint himself with the reasons for many steps in his work.

I lay no claim to originality of methods presented, but rather have brought together a compilation of the methods as used in laboratory work during several years of experience, and such as have been found to give excellent satisfaction in the hands of my own students.

If the methods herein presented prove as successful in the hands of others as in the hands of my own students, and they are drawn to a further study and a better understanding of the products of nature, the hopes of its author will be more than realized.

AGRICULTURAL COLLEGE, FARGO, N. D.,  
*October, 1897.*

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# QUANTITATIVE CHEMICAL ANALYSIS.

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## CHAPTER I.

### INTRODUCTION.

QUANTITATIVE ANALYSIS may be subdivided into two divisions, according to the methods employed—gravimetric and volumetric analysis. In gravimetric analysis the constituents to be determined are converted into compounds of definite composition, which by methods of filtration and washing may be separated from all others, and after further and appropriate treatment be weighed. From the known composition of the product as weighed, the amount of the unknown constituent may be calculated.

In volumetric analysis the constituent is determined, not by weighing a known product, but rather by means of well-known reactions brought about by means of solutions of a definite and known strength. To determine accurately, then, the amount of the known solution added, is the essential in volumetric analysis.

Volumetric analysis is quantitative analysis by measure, while gravimetric analysis is quantitative analysis by weight.

#### GENERAL DIRECTIONS.

1. Cleanliness and neatness in every detail is an absolute essential to success. The work-desk should be kept neat and clean, using freely a sponge, which should always be at hand.

2. Protect all preparations and solutions from dust, from splatterings, and from all unnecessary fumes. See that every piece of apparatus is washed and wiped dry after using, and examine each piece before using, to see that it is perfectly clean.

3. Read each experiment through carefully before attempting to undertake the work, and be sure you understand every step in the operation, and are able to answer all the questions asked. See that all the necessary apparatus is at hand before beginning, and have everything in place and properly arranged.

4. No student can make any degree of success who does not keep a neat and legible note-book, in which every detail is written down as soon as observed. All weights and measures should be entered as soon as made, and not kept on a slip of paper to be written up at some future time. One of the greatest shortcomings of every young experimenter or chemist is a

lack of proper notes on work done. Remember that what is perfectly clear to you to-day may be forgotten by you to-morrow, or next year, when the facts may be needed.

5. Every piece of apparatus used in the experiment or work should be so labelled as to be readily identified at any time, and no mistake made in the work.

6. In dissolving or neutralizing substances that effervesce, always add the substance slowly to prevent loss either from frothing or from so rapid effervescence as to lose the material by spattering over the sides of the dish.

#### DESICCATORS.

Desiccators should contain a little concentrated sulfuric acid, which from time to time may be renewed. The cover of the desiccator should be made air-tight by applying about the edges a thin coating of vaseline.

Some chemists prefer for general use fused anhydrous calcium chlorid, nearly filling the desiccator with this product. The chlorid needs renewing frequently, and should not be allowed to cake.

The desiccator should never be allowed to remain uncovered.

#### FILTERING, WASHING, AND BURNING.

1. Select a funnel of proper size for the work in hand, fold the filter, carefully place in funnel, and

unless otherwise mentioned moisten the filter with distilled water, and see that the paper fits nicely against all sides of the funnel. This last is essential for success.

2. The paper in the funnel should not come within one-fourth inch of top of funnel. If paper reaches top of funnel the precipitate cannot be properly washed.

3. In pouring solutions into funnel, always use a glass rod, pouring the solution down its side.

4. *Washing*.—Never add wash-water to the contents of the funnel until all the solution has filtered through, then direct the stream of water against the side of the funnel, above the paper, and gradually bring it down, washing the precipitate down toward the centre of the paper. See that all the wash-water is run through before adding a fresh portion. To remove the last particles of adhering precipitate from the beaker, use a glass rod with a small piece of rubber tubing at the end. This rubber-tipped end of the rod must not be used in the solution, and only in the wash-water.

5. *Drying*.—Precipitates to be dried in funnel should be covered with glazed paper neatly folded around the edge of the funnel to keep it in place, and then small holes perforated through the paper to permit drying. These may then be placed in water-oven and allowed to remain until dry. See that each



funnel is properly labelled before removing it from its stand.

6. *Burning*.—Place the weighed crucible upon a piece of glazed paper. Remove the filter and contents from the funnel, unfold the filter, and remove carefully all the precipitate, transferring it to the crucible; then fold the filter-paper in, and coil a small platinum wire about it, holding it carefully by the wire over the crucible on the glazed paper, and set the paper on fire, holding above it a funnel so as to form an upward current and to catch any particles that might be carried off mechanically. If the ash of the paper needs further burning, it may now be placed upon the crucible cover and burned, when it should be carefully transferred to the crucible with the main precipitate, and all particles from the glazed paper united with it. When the heat is first applied to the crucible the flame should be moved about, to warm all parts uniformly, to prevent breaking the porcelain or injuring the platinum dish.

#### WEIGHING.

1. Always test the balance before each weighing to see that it is in perfect adjustment. If the swing of the needle is not the same to each side of the zero, then the balance should be brought into adjustment before attempting to weigh.

The student should never attempt to make adjustment of the balance, but call upon the instructor.

2. The balance should be brushed off and kept clean. Salts or liquids should not be allowed to come in contact with the pans.

3. When not in use, return the weights to their proper place in the weight-box. Make two readings of weights. First read the weights missing from weight-box, then return the weights from the pan to their proper place, reading them again. In this way the student has a check upon his weighings.

4. The balance-case should always be closed during the final weighings before using the rider, and thus protect the pans from any currents of air.

5. Never trust to memory the weights of substances, but make a memorandum of all weighings at the time of taking the readings of the weights from the pan.

#### CARE OF PLATINUM.

1. Never heat the platinum dish in contact with iron. Use only platinum or pipe-stem triangles.

2. Do not fuse pure metals in a platinum crucible or salts of the silver, arsenic, copper, and zinc groups.

3. Do not fuse the sulfid of metals in platinum.

4. Do not use aqua regia or free chlorin on platinum ware.

5. Alkaline nitrates, hydrates, or cyanids should not be fused in platinum.

6. Keep the dish always perfectly bright and clean, and for this purpose there is nothing better than fine sea-sand free from the sharp grit. Use only the moistened finger for applying the same. Occasionally fusing with a little  $\text{KHSO}_4$  and then carefully polishing with sand will be found a good method for cleaning badly tarnished platinum ware.

7. Never allow a smoky flame to be used with platinum, or if the reducing flame comes in contact with the platinum there is soon formed a gray film, and the platinum is injured.

#### SOLUTIONS.

Nearly all the solutions required are mentioned in connection with the method of analysis. A few stock solutions may be required, the methods of preparation of which are given here.

*Molybdate Solutions.*—Dissolve 100 grams of molybdic acid in 417 c.c. of ammonia, sp. gr. 0.96. Pour the solution into 1250 c.c. of nitric acid, sp. gr. exactly 1.20. The solutions should be united slowly, and always in the order given above. Let the mixture stand for several days in a warm place and then decant the solution from the sediment and preserve for use.

*Magnesia Mixture.*—Of recently ignited  $\text{MgO}$ , dissolve 22 grams, or 110 grams crystallized  $\text{MgCl}$ , in dilute  $\text{HCl}$ , avoiding any excess. When dissolved add a slight excess of calcined magnesia and boil, to precipitate iron, and then filter and add 280 grams of ammonium chlorid, 700 c.c. of  $\text{NH}_4\text{OH}$ , sp. gr. 0.96, and make up to 2000 c.c. with distilled water.

## CHAPTER II.

### GRAVIMETRIC ANALYSIS.

#### 1. Iron (Fe ; 56).

WEIGH out 0.3 gram pure iron wire, place in beaker of about 250 c.c. capacity, add water and nitric acid, heat slowly to dissolve, avoid much excess of  $\text{HNO}_3$ , and to the hot solution add  $\text{NH}_4\text{OH}$  slowly and with constant stirring until slightly alkaline; boil for a few minutes, allow ppt. to settle, filter, and wash thoroughly with hot distilled water. Dry this ppt. thoroughly in the steam-oven, then transfer ppt. to a tared porcelain crucible; burn filter-paper and add ash to crucible, cover the crucible and ignite, at first slowly, then intensely, with blast-lamp; cool in desiccator, and weigh as  $\text{Fe}_2\text{O}_3$ .

*Formula.*— $\text{Fe}_2\text{O}_3 : \text{Fe} :: \text{ppt.} : x$ ;

$x$  = amount of Fe in ppt

$*M : x :: 100 : y$ ;

$y$  = per cent Fe.

---

\*  $M$  in all cases indicates the mass or amount of original substance weighed out.

*Questions.*—Why dry ppt. before igniting? Why add  $\text{HNO}_3$ ? Is there any danger of loss by volatilizing the iron?

## 2. Magnesium (Mg; 24).

Of magnesium foil or powder weigh out about .15 gram, add water and  $\text{HCl}$  slowly to dissolve the  $\text{Mg}$ , heat to bring all into solution, then add  $\text{NH}_4\text{Cl}$  and  $\text{NH}_4\text{OH}$  in slight excess. If ppt. forms add more  $\text{NH}_4\text{Cl}$  till redissolved. To this solution add  $\text{Na}_2\text{HPO}_4$ , as long as ppt. forms, stir carefully without touching sides of beaker, and allow to stand until the solution above ppt. is perfectly clear. Filter and wash out beaker with portions of the filtrate, and then wash with dilute ammonia water (1 pt.  $\text{NH}_4\text{OH}$  to 3 pts.  $\text{H}_2\text{O}$ ) until filtrate gives no coloration with  $\text{HNO}_3$  and  $\text{AgNO}_3$ . Dry thoroughly, remove ppt. to tared porcelain crucible, burn filter-paper and add the ash to the crucible and its contents, and ignite, at first slowly, then to intense heat; cool, and weigh as  $\text{Mg}_2\text{P}_2\text{O}_7$ .

*Formula.*— $\text{Mg}_2\text{P}_2\text{O}_7 : \text{Mg}_2 :: \text{ppt.} : x$ ;

$x$  = amount of  $\text{Mg}$  in ppt.

$M : x :: 100 : y$ ;

$y$  = per cent  $\text{Mg}$ .

*Questions.*—Why add  $\text{HNO}_3$ ? Why not ignite in platinum crucible? Why add  $\text{NH}_4\text{OH}$ ? Why wash with  $\text{NH}_4\text{OH}$  water? What is composition of ppt.?

### 3. Aluminum (Al; 27).

Weigh out about one gram dry alum  $\text{AlK}(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$  and dissolve in about 100 c.c. distilled water; add 25 c.c.  $\text{NH}_4\text{Cl}$ , then  $\text{NH}_4\text{OH}$  to slight alkalinity, avoiding much excess, and heat to drive off any excess of  $\text{NH}_4\text{OH}$ . Allow to stand until ppt. subsides, and then filter and wash thoroughly; dry, transfer ppt. to tared platinum crucible, ignite filter-paper, add ash, ignite carefully, and finally with full blast; cool, and weigh as alumina,  $\text{Al}_2\text{O}_3$ .

*Formula.*— $\text{Al}_2\text{O}_3 : \text{Al}_2 :: \text{ppt.} : x$ ;

$x$  = amount Al in ppt.

$M : x :: 100 : y$ ;

$y$  = per cent Al.

*Questions.*—Why add  $\text{NH}_4\text{Cl}$ ? Why avoid excess of  $\text{NH}_4\text{OH}$ ? Why ignite intensely at close? What is composition of ppt.?

### 4. Calcium (Ca; 40).

Weigh out about 0.5 grams  $\text{CaCO}_3$ , add 50 c.c.  $\text{H}_2\text{O}$ , and slowly  $\text{HCl}$ , and heat to dissolve Ca and to drive off excess of acid; add  $\text{NH}_4\text{OH}$  until slightly alkaline and then  $(\text{NH}_4)_2\text{C}_2\text{O}_4$  to ppt. all the Ca; allow to stand twelve hours, filter, and wash with hot water containing a little ammonia; dry, and transfer ppt. to

tared platinum crucible; burn filter-paper, add ash to the crucible, and ignite, at first slowly and finally with full blast; cool, and weigh as CaO.

*Formula.*— $M : \text{ppt.} :: 100 : x$ ;  
 $x = \text{per cent CaO.}$

By igniting at low heat the ppt. may be weighed as  $\text{CaCO}_3$ , then converted into CaO it may then be dissolved in HCl, and  $\text{H}_2\text{SO}_4$  added to form  $\text{CaSO}_4$ , and determined in this form.

*Questions.*—What is the composition for the first ppt. of Ca? What change takes place in crucible by heating ppt. at low heat? With intense heat? Why not weigh as  $\text{CaCO}_3$ ? Why was  $\text{NH}_4\text{OH}$  added? Why add  $\text{NH}_4\text{OH}$  to wash-water? Why not filter at once? Why have solution hot when  $(\text{NH}_4)_2\text{C}_2\text{O}_4$  is added?

### 5. Barium (Ba; 137).

Weigh out about 1 gram barium chlorid that has been well dried, add 150 c.c. water, 1 c.c. HCl, and heat to drive off excess of acid. To the hot solution add diluted  $\text{H}_2\text{SO}_4$  until slightly acid, and with constant stirring keep near boiling for ten minutes; then allow ppt. to settle and decant on to a firm filter-paper. To the ppt. add boiling water and a few drops of  $\text{H}_2\text{SO}_4$ , then decant, add hot water to the ppt., and filter, washing carefully with boiling water



on the filter until filtrate gives no turbidity with  $\text{BaCl}_2$ . Dry ppt. and then remove it to tared platinum crucible; burn filtrate carefully, putting ash on crucible; cover, adding a drop of  $\text{H}_2\text{SO}_4$ , and ignite; then transfer ash to crucible, and heat low for some time, and finally raise temperature to low redness; cool, and weigh as barium sulfate.

*Formula.*— $\text{BaSO}_4 : \text{Ba} :: \text{ppt.} : x$ ;

$x$  = amount of Ba in ppt.

$M : x :: 100 : y$ ;

$y$  = per cent Ba.

*Questions.*—Why must there be an excess of  $\text{HCl}$ ? Why remove ppt. from filter before igniting? Why ignite at low red heat? Why have solution dilute before adding precipitant?

## 6. Sulfuric Acid ( $\text{SO}_3$ ; 80).

Weigh out about one gram copper sulfate, and add 100 c.c. water, a few drops of  $\text{HCl}$ , and 20 c.c.  $\text{NH}_4\text{Cl}$ ; then heat to boiling, remove lamp, and add drop by drop  $\text{BaCl}_2$  until in slight excess, stirring constantly. Allow to stand in warm place for twelve hours. Decant through filter, add boiling water to ppt. and a few drops of  $\text{H}_2\text{SO}_4$ ; boil, decant; add more boiling water, filter, and wash thoroughly. Be sure to allow each portion of wash-water to pass through

before adding more. Dry ppt., transfer to tared crucible, burn, filter, and moisten ash with  $\text{H}_2\text{SO}_4$  on crucible cover; dry and ignite, then transfer ash to crucible, and heat very low and finally at dull red heat for ten minutes; cool, and weigh as  $\text{BaSO}_4$ .

*Formula.*— $\text{BaSO}_4 : \text{SO}_3 :: \text{ppt.} : x$ ;

$x$  = amount  $\text{SO}_3$  in ppt.

$M : x :: 100 : y$ ;

$y$  = per cent  $\text{SO}_3$ .

*Questions.*—Why acidify with  $\text{HCl}$ ? Why have solution hot when  $\text{BaCl}_2$  is added? Why add  $\text{HN}_4\text{Cl}$ ? Why not burn ppt. and paper together? Why is such extreme care in details necessary?

## 7. Chlorin ( $\text{Cl}$ ; 35.4).

Weigh out about .25 gram  $\text{NaCl}$  that has been heated to expel moisture, dissolve in 75 c.c. water, and add slowly  $\text{AgNO}_3$  containing a few drops of  $\text{HNO}_3$ ; heat nearly to boiling and stir vigorously, and continue until ppt. settles, leaving liquid above clear; filter, wash with hot water containing a few drops of  $\text{HNO}_3$  until wash-water gives no turbidity with  $\text{HCl}$ ; dry ppt., transfer all of ppt. to tared porcelain crucible, burn filter-paper carefully, and add ash to crucible, then a few (four) drops of  $\text{HNO}_3$  and a drop or two of  $\text{HCl}$ ; dry, ignite very gently, moving burner

about under crucible until ppt. just begins to fuse about the edges of crucible; then cool and weigh, avoiding direct sunlight.

*Formula.*— $\text{AgCl} : \text{Cl} :: \text{ppt.} : x$ ;  
 $x = \text{amount of Cl in ppt.}$   
 $M : x :: 100 : y$ ;  
 $y = \text{per cent Cl.}$

*Questions.*—Why not use platinum crucible? Why not add  $\text{HNO}_3$ ? Why avoid sunlight? Why ignite so low?

### 8. Copper (Cu; 63.2).

Weigh out about one gram of well-desiccated copper sulfate, add 100 c.c. hot water and heat to boiling. The solution should be clear, and to the hot solution add drop by drop a dilute solution of  $\text{NaOH}$ , using but slight excess of reagent. Stir constantly, and continue to heat for ten minutes; allow ppt. to subside, and then decant on to filter; add water, and boil ppt. again. Filter, and wash thoroughly with hot water, dry in steam-oven, transfer ppt. completely to tared crucible, burn filter, add ash, and ignite intensely; cool, and weigh as oxid.

*Formula.*— $\text{CuO} : \text{Cu} :: \text{ppt.} : x$ ;  
 $x = \text{amount of Cu in ppt.}$   
 $M : x :: 100 : y$ ;  
 $y = \text{per cent Cu.}$

Should the ppt. adhere firmly to sides of beaker it may be dissolved off with a few drops of  $\text{HNO}_3$ , precipitated again with  $\text{NaOH}$  and added to filter.

*Questions.*—Why decant and boil with  $\text{H}_2\text{O}$  instead of filtering at once? Why heat to boiling before adding  $\text{NaOH}$ ? Why use but slight excess of reagent?

### 9. Lead ( $\text{Pb}$ ; 207).

Weigh out about one gram  $\text{Pb}(\text{NO}_3)_2$ , dissolve in 75 c.c. water, and add slowly  $\text{H}_2\text{SO}_4$  in slight excess, and then add an equal volume of alcohol; mix well and allow to stand for twelve hours; filter, wash with alcohol (3 volumes alcohol and 1 volume  $\text{H}_2\text{O}$ ), dry, and remove carefully all the ppt. to a tared crucible. Burn filter, moisten with  $\text{HNO}_3$  and  $\text{H}_2\text{SO}_4$ , dry and ignite, and transfer the well-burned ash from crucible cover to crucible with ppt., and heat only to low redness; cool and weigh. Observe that the reducing flame does not come in contact with ppt.

*Formula.*— $\text{PbSO}_4 : \text{Pb} :: \text{ppt.} : x$ ;

$x$  = amount of  $\text{Pb}$  in ppt.

$M : x :: 100 : y$ ;

$y$  = per cent  $\text{Pb}$ .

*Questions.*—Why burn filter completely before uniting with ppt.? Why wash with alcohol? Will the presence of  $\text{NH}_4\text{OH}$  salts or  $\text{HNO}_3$  interfere in this

method? Why? How prevented? Why add alcohol before adding reagent?

### 10. Manganese (Mn; 55).

Weigh out about .5 gram  $\text{MnCl}_2$ , dissolve in 100 c.c. of water, add a few drops  $\text{HCl}$ , then  $\text{NH}_4\text{OH}$  in excess, then  $\text{Na}_2\text{HPO}_4$  in large excess; dissolve the white ppt. with  $\text{H}_2\text{SO}_4$ , heat to boiling, and add  $\text{NH}_4\text{OH}$  in excess. Place on water-bath for one hour, when the ppt. forms crystalline scales and is rose-colored. Filter, and wash with hot water containing a little  $\text{NH}_4\text{OH}$ . Dry, and transfer to tared porcelain crucible, adding ignited filter ash, and burn at bright red heat, when the ppt. is converted into a nearly white powder,  $\text{Mn}_2\text{P}_2\text{O}_7$ .

*Formula.*— $\text{Mn}_2\text{P}_2\text{O}_7 : \text{Mn}_2 :: \text{ppt.} : x$ ;

$x$  = amount of Mn in ppt.

$M : x :: 100 : y$ .

$y$  = per cent Mn.

*Questions.*—Why redissolve ppt. with  $\text{H}_2\text{SO}_4$ ? Why add to solution  $\text{NH}_4\text{OH}$ ? Why heat for one hour? Is ppt. soluble in  $\text{H}_2\text{O}$ ?

### 11. Zinc (Zn; 65).

Dissolve about one gram pure zinc sulfate in 100 c.c.  $\text{H}_2\text{O}$  in porcelain dish, heat to boiling, and add

$\text{Na}_2\text{CO}_3$  slowly, with stirring, until an excess of reagent is added. Allow to settle and decant through filter, and boil the ppt. three times with water and decanting of each separate portion of water; finally, transfer ppt. to filter, and wash with hot water. Dry, transfer ppt. to crucible, burn ash, add to ppt., and ignite gently at first, and finally to full redness; cool, and weigh as  $\text{ZnO}$ .

*Formula.*— $\text{ZnO} : \text{Zn} :: \text{ppt.} : x$ ;

$x$  = amount of Zn in ppt.

$M : x :: 100 : y$ ;

$y$  = per cent Zn.

*Questions.*—Why use porcelain dish in place of beaker? What is composition of ppt.?

## 12. Phosphoric Acid ( $\text{P}_2\text{O}_5$ ; 142).

Weigh out about .5 gram ammonium phosphate well dried, dissolve in 75 c.c.  $\text{H}_2\text{O}$ , add 12 c.c.  $\text{NH}_4\text{Cl}$ , and then “magnesia mixture” slowly with constant stirring, but do not touch sides of beaker until an excess of reagent is added. Allow to stand fifteen minutes and add 25 c.c.  $\text{NH}_4\text{OH}$ , and stand in warm place for about twelve hours. Decant on to filter, using some of filtrate for purpose if necessary, and wash well with ammonia water (1 to 3) until washing gives no reaction for Cl. Dry ppt. and treat the same as in Mg, weighing as  $\text{Mg}_2\text{P}_2\text{O}_7$ .

*Formula.*— $\text{Mg}_2\text{P}_2\text{O}_7 : \text{P}_2\text{O}_5 :: \text{ppt.} : x;$

$x$  = amount of  $\text{P}_2\text{O}_5$  in ppt.

$M : x :: 100 : y;$

$y$  = per cent  $\text{P}_2\text{O}_5$ .

*Questions.*—Why add  $\text{NH}_4\text{Cl}$ ? What is “magnesia mixture”? Why not let rod touch sides of beaker? Why add  $\text{NH}_4\text{OH}$  after adding reagent? How shall we test for Cl in wash-water, and why?

### 13. Potassium ( $\text{K}_2\text{O}$ ; 94).

Weigh out about .3 gram of potassium chlorid, the salt having been previously heated and cooled in a desiccator. Dissolve in porcelain evaporating-dish with as little water as convenient (5 to 8 c.c.), add platinic chlorid to slight excess, and evaporate almost to dryness on water-bath; then add 40 c.c. 80 per cent alcohol, and allow to stand for one hour; then decant on weighed filter and add fresh alcohol, and repeat two or three times, and then transfer to filter and wash with ether; dry, cool in desiccator, and weigh. From total weight deduct weight of filter-paper, or a Gooch crucible with asbestos filter may be used. Ppt. is  $\text{K}_2\text{PtCl}_6$ . In place of alcohol a mixture of alcohol and ether is preferable (2 to 1).

*Formula.*— $\text{K}_2\text{PtCl}_6 : \text{K}_2\text{O} :: \text{ppt.} : x;$

$x$  = amount of  $\text{K}_2\text{O}$  in ppt.

$M : x :: 100 : y;$

$y$  = per cent  $\text{K}_2\text{O}$ .

*Questions.*—Why use mixture of alcohol and ether? Why not use absolute alcohol? May K be determined as a sulfate? Why not ignite ppt.?

#### 14. Sodium ( $\text{Na}_2\text{O}$ ; 62).

Weigh out of  $\text{Na}_2\text{CO}_3$ , about .4 gram; dissolve in water in tared porcelain dish, add slowly a slight excess of HCl, evaporate to dryness on water-bath, moisten with HCl, dry, and then heat gently at  $110^\circ$  for ten minutes; then bring heat up till dull redness, moving the flame about constantly to all parts of dish; cool, and weigh as sodium chlorid.

*Formula.*— $2\text{NaCl} : \text{Na}_2\text{O} :: \text{ppt.} : x$ ;

$x$  = amount  $\text{Na}_2\text{O}$  in ppt.

$M : x :: 100 : y$ ;

$y$  = per cent  $\text{K}_2\text{O}$ .

*Questions.*—Why heat ppt. so carefully at first? Why moisten ppt. with HCl and evaporate? Why not heat to convert direct into  $\text{Na}_2\text{O}$ ?

#### 15. Separation of K and Na.

Weigh out .5 gram of mixed salts, dissolve in 50 c.c. water, add HCl to the solution, then  $\text{Ba}(\text{OH})_2$ , as long as ppt. forms; boil for three minutes, filter while hot, and wash with boiling water. To filtrate add a



few drops of  $\text{NH}_4\text{OH}$  and then  $(\text{NH}_4)_2\text{CO}_3$  to ppt. Ba; filter, and evaporate to dryness in porcelain dish, gradually raising heat to dull redness to drive off ammonia salts. Dissolve with 15 c.c.  $\text{H}_2\text{O}$ , add a few drops of  $\text{NH}_4\text{OH}$  if ppt. forms, filter into tared dish, wash, and evaporate to dryness; heat to dull redness, cool, and weigh total alkalis as chlorids. Dissolve residue with 10 c.c. water and add platinic chlorid as in K determination, precipitating K and Na. After evaporating dissolve out Na with alcohol, leaving  $\text{K}_2\text{PtCl}_6$  to be treated and weighed as in potash.

*Formula for  $\text{K}_2\text{O}$ .—* $\text{K}_2\text{PtCl}_6 : \text{K}_2\text{O} :: \text{ppt.} : x$ ;

$x$  = amount  $\text{K}_2\text{O}$  in ppt.

$M : x :: 100 : y$ ;

$y$  = per cent  $\text{K}_2\text{O}$ .

*Formula for  $\text{Na}_2\text{O}$ .—* $\text{K}_2\text{PtCl}_6 : 2\text{KCl} :: \text{ppt.} : x$ ;

$x$  = amount KCl in total alkalis.

Total alkalis —  $x$  = ppt. as NaCl.

$2\text{NaCl} : \text{Na}_2\text{O} :: \text{amount NaCl} : x$ ;

$x$  = amount  $\text{Na}_2\text{O}$  in ppt.

$M : x :: 100 : y$ ;

$y$  = per cent  $\text{Na}_2\text{O}$ .

*Questions.*—Why weigh ppt. first as chlorids? How was Na separated from K? Why not evaporate solution fully to dryness after adding platinic chlorid?

**16. Carbonic Acid ( $\text{CO}_2$ ; 44).**

A. Weigh into a tared platinum crucible about 1.5 grams well dried and pulverized marble, mix with about four times its weight of powdered borax, and fuse. Loss is taken as  $\text{CO}_2$ . This method is not applicable in many cases, and is at best only an approximation.

B. About one gram of the carbonate is placed in a flask of 200 c.c. capacity, fitted with a rubber stopper having three perforations. Through one perforation is passed a small separatory funnel containing dilute  $\text{HCl}$ . A second opening has a small glass tube connected with a U tube containing  $\text{KOH}$  solution, and this connected with another tube having  $\text{H}_2\text{SO}_4$ . These tubes and reagents are for drying the air that may be drawn through the apparatus during the analysis. The third opening has a bent glass tube connected with a U tube containing concentrated  $\text{H}_2\text{SO}_4$  to serve as a drying agent for evolved gas; this is attached to a  $\text{KOH}$  bulb containing strong  $\text{KOH}$  solution (1 to 1), and to the end of this is attached a small U tube containing  $\text{H}_2\text{SO}_4$  to prevent any moisture from entering the  $\text{KOH}$  bulb, and the whole attached to a suction-pump. (In place of the pump one may use a large bottle, properly arranged, filled with water and inverted.) When ready for starting, the  $\text{KOH}$  bulb should be carefully weighed and again attached

in the circuit, and the dilute HCl allowed to flow drop by drop into the flask upon the marble or other carbonate, and when the decomposition is complete the flask, on a sand-bath, should be gently heated, and the suction-pump started very slowly, drawing through a continuous stream of air,  $\text{CO}_2$ , free. When all the gas in the apparatus has been drawn through the KOH bulb the pump may be shut off, the bulbs removed and again weighed. The increase in weight is due to  $\text{CO}_2$ . This method is very accurate and reliable if the work is carefully done and all leaks avoided.

*Questions.*—Why use  $\text{H}_2\text{SO}_4$  in U tubes? Why is not method A satisfactory?

### 17. Dolomite.

Of the well pulverized and dried mineral weigh out about one gram, put into porcelain dish, add a little water, and then HCl with a few drops of  $\text{HNO}_3$ . Evaporate to dryness, moisten with HCl, add water, and heat on water-bath; filter, wash with hot water, dry, and ignite residue as  $\text{SiO}_2$ .

*Iron and Alumina.*—To the filtrate above add  $\text{NH}_4\text{Cl}$  and  $\text{NH}_4\text{OH}$  until alkaline, heat to drive off excess of  $\text{NH}_3$ ; filter, wash with hot water, dry, ignite, cool, and weigh as  $\text{Al}_2\text{O}_3$  and  $\text{Fe}_2\text{O}_3$ .

*Lime.*—To the filtrate above add  $\text{NH}_4\text{OH}$  and  $(\text{NH}_4)_2\text{C}_2\text{O}_4$ . The completion of the determination is the same as for Ca, which see.

*Magnesia*.—To the above filtrate add  $\text{Na}_2\text{HPO}_4$  and  $\text{NH}_4\text{OH}$  and treat ppt. as in magnesium, which see.

*Volatile Matter*.—Weigh out in tared platinum crucible about .5 gram of dolomite; heat intensely until no further loss in weight. Loss is  $\text{CO}_2$  and other volatile matter.

### 18. Feldspar (Orthoclase and Albite).

Of the finely pulverized and dried mineral weigh out about one gram, add 13 grams of a mixture in equal parts of potassium and sodium carbonates and fuse in platinum crucible, and cool quickly. Place the crucible in porcelain dish, boil with  $\text{H}_2\text{O}$  and  $\text{HCl}$  in excess, evaporate on water-bath to dryness, and heat at  $120^\circ$  for one hour, to render  $\text{SiO}_2$  wholly insoluble. Moisten the dry mass with  $\text{HCl}$ , add  $\text{H}_2\text{O}$ , heat to boiling for ten minutes, filter, and wash. The residue is dried, ignited, cooled, and weighed as  $\text{SiO}_2$ .

Determine iron, alumina, lime, and magnesia in filtrate in same manner as in dolomite.

The alkalis must be determined in a separate portion, as follows: Weigh out about .5 gram of the mineral, add .5 gram  $\text{NH}_4\text{Cl}$  and four grams of calcium carbonate, and heat in platinum crucible the well-mixed mass at first gently and then at low redness for one and one-half hours; then cool and put crucible in porcelain dish, add  $\text{H}_2\text{O}$ , and boil for twenty minutes, replacing water from time to time; filter, and to

filtrate add HCl and evaporate to dryness and heat residue for some time at  $120^{\circ}$ ; pulverize residue in mortar, add  $H_2O$ , boil, filter, and wash into the filtrate. Determine alkalis as for separation of K and Na, which method see.

### 19. Gypsum.

*Moisture and Water of Crystallization.*—Of the well-pulverized material weigh out in platinum crucible about 2.5 grams, add a little  $(NH_4)_2CO_3$ , and gradually heat to  $150^{\circ}$  to drive off all the ammonia salts; cool and weigh. Loss in weight is calculated as moisture and water of crystallization.

Weigh out about one gram of the finely pulverized gypsum, add HCl, and heat on water-bath (50 c.c.  $H_2O$  and 10 c.c. strong HCl). Continue the digestion for one hour or more, then filter, using a weighed filter; wash, dry, and weigh filter and residue; then ignite and weigh again, calculating loss as organic matter and the residue as clay and sand.

Make the filtrate up to 200 c.c.; divide into two parts A and B.

*Sulfuric Acid.*—Nearly neutralize A with  $Na_2CO_3$ , heat solution nearly to boiling and add  $BaCl_2$ , and proceed as under  $H_2SO_4$  determination. From the sulfuric acid calculate the per cent of gypsum.

*Iron and Alumina.*—To B add  $HNO_3$  to oxidize

any iron from ferrous to ferric condition, and determine iron and alumina as in dolomite.

*Lime.*—To the above filtrate heated to boiling add  $(\text{NH}_4)_2\text{C}_2\text{O}_4$ , and determine lime as in dolomite.

*Carbonic Acid.*—In one gram of the finely-pulverized gypsum determine the  $\text{CO}_2$  as under carbonic acid, and from this calculate the per cent of calcium carbonate contained in the gypsum.

## 20. Mineral Phosphates.

*Apatite.*—Pulverize finely and dry to  $100^\circ$ . Of the dry mineral weigh out one gram in a porcelain dish, add 100 c.c. concentrated  $\text{HCl}$  and evaporate to dryness on water-bath; moisten residue with  $\text{HCl}$ , add 10 c.c. of dilute  $\text{HNO}_3$ , and evaporate again to dryness. To the residue add 50 c.c. water and 5 c.c.  $\text{HNO}_3$ , filter, and make up to 200 c.c. To 50 c.c. of filtrate add about 15 grams dry ammonium nitrate, heat, add 50 c.c. of "molybdate solution," and keep at a temperature of  $65^\circ$  for one hour or in a warm place at about  $60^\circ$  for several hours. Filter and wash with a solution of ammonium nitrate. Dissolve the ppt. on the filter with ammonia and hot water, nearly neutralize solution with  $\text{HCl}$ , cool, add "magnesia mixture" slowly, and treat as in previous determination of  $\text{P}_2\text{O}_5$ .

The determination of bases may be made as in dolomite and feldspar if desired.

## CHAPTER III.

### VOLUMETRIC ANALYSIS.

BRIEFLY, volumetric analysis is quantitative analysis by measure instead of by weight, as in gravimetric. For volumetric work, three things are essential:

- 1st. Solutions, the chemical power of which is absolutely known—called “Standard Solutions.”
- 2d. Carefully graduated vessels called “burettes.”
- 3d. A means of detecting by the eye the end reaction. This is usually made possible by means of an indicator.

There are also three important classes of reactions to be considered:

1. Where the determination is effected by saturation or by neutralization with substances of opposite chemical properties, as acids and alkalis.
2. Where the determination is made by processes of reduction or oxidation, using agents of known power, as potassium permanganate, potassium bichromate, iodine, etc., as oxidizing agents, and ferrous and stannous compounds and sodic thiosulfate as reducing agents.

3. Where the determination is effected by precipitation in some insoluble and definite combination, as in the case of silver and chlorin.

Many determinations are best made wholly or in part by means of volumetric methods. For this purpose standard solutions are necessary, and they are usually made of such strength that 1000 c.c. would contain an amount exactly equivalent to one gram of hydrogen, and are known as normal solutions; that is, are of such strength that 1 c.c. of any of the acids will exactly neutralize 1 c.c. of any of the bases. Such solutions are made by weighing the equivalent of the molecular weight of univalent acids and bases, or one-half of the molecular equivalent of divalent acids or bases, and bringing the solution to 1000 c.c. by the addition of distilled water. Semi-normal would be half this strength, tenth normal ( $\frac{1}{10}$ ) one-tenth this strength, and others in the same manner. A normal solution of NaOH would contain 40 grams NaOH per 1000 c.c., and would just neutralize an equal amount of normal HCl solution containing 36.37 grams HCl. For volumetric work the first essential is to prepare accurately adjusted normal solutions, and these must be kept in well-stoppered bottles, and best in a cool place. From time to time they need to be restandardized to see that the "titre" remains unchanged,



## 21. Normal Sulphuric Acid.

Weigh out 60 grams of concentrated  $\text{H}_2\text{SO}_4$ , and add this to 1050 c.c. of distilled  $\text{H}_2\text{O}$ ; mix well, and allow to cool to about  $18^\circ \text{C}$ ., at which temperature all volumetric determinations should be made. Determine, by means of barium chlorid, the amount of sulfuric acid in at least three portions each of exactly 20 c.c. The results must agree, or further determinations will be necessary, and the average of all concordant determinations taken as the mean. Determine from this mean the amount of water necessary to add, so that each 1000 c.c. shall contain exactly 49 grams of  $\text{H}_2\text{SO}_4$ , or 40 grams of  $\text{SO}_3$ , which is the half molecular weight. Thus if 1.078 grams  $\text{H}_2\text{SO}_4$  were found in 20 c.c., then in 1000 c.c. there would be 53.9 grams of  $\text{H}_2\text{SO}_4$ , in place of 49 grams, as should be for a normal solution; therefore every 1000 c.c. of the solution should be diluted to 1100 c.c. ( $49 : 1000 :: 53.9 : x = 1100 \text{ c.c.}$ ). One c.c. of this solution contains .049 gram  $\text{H}_2\text{SO}_4$ . From the solution by dilution N/5, N/10, or N/100 solutions may be prepared at any time.

## 22. Normal ( $\text{Na}_2\text{CO}_3$ ).

Ignite to low redness chemically pure neutral sodium carbonate and from the desiccated product weigh out 53 grams and dissolve to one litre. One

c.c. of this should exactly neutralize 1 c.c. of the normal  $\text{H}_2\text{SO}_4$ . If it does not, then one of the solutions is wrong, and an effort should be made to determine the source of the error.

### 23. Semi-normal ( $\text{NH}_3$ ).

A normal solution of  $\text{NH}_3$  does not keep its strength well, but a half-normal solution gives good satisfaction, and should be of such strength that each 1000 c.c. shall contain 8.5 grams  $\text{NH}_3$ . This is made by diluting ammonia with distilled water until 20 c.c.  $\text{NH}_3$  will be exactly neutralized by 10 c.c. of the normal  $\text{H}_2\text{SO}_4$  or 20 c.c. of the half normal, using cochineal, litmus, or phenol-phthalein solutions as an indicator.

### 24. Indicators.

Cochineal is prepared by digesting  $1\frac{1}{2}$  grams pulverized cochineal in 25 c.c. alcohol and 100 c.c. water for a few days at ordinary temperature, with occasional shaking. The filtered solution is used, and has the advantage of not being acted upon by  $\text{CO}_2$  to the extent of the other indicators, but it cannot be used in presence of iron salts.

*Litmus* is prepared by digesting one part of the litmus in six of distilled water; filter, and divide solution into two parts; to one part add  $\text{HNO}_3$  until of reddish color, and then mix the two solutions and

add one part of strong alcohol. Litmus cannot be used in presence of carbonic acid.

*Phenol-phthalein* is best prepared by dissolving one gram of phenol-phthalein in 100 c.c. of 95 per cent alcohol. With the aid of the above normal solutions the strength of various alkaline or acid solutions may be readily determined, and the same solutions can be used in food analysis, and wherever nitrogen is to be determined as ammonia.

## 25. Iron Determination.

*Standard Decinormal Potassium Permanganate* is made by weighing out of the dry and pure salt 3.156 grams and dissolving to 1000 c.c. The solution must be kept in a cool, dark place. In titrating permanganate there must be present free acid, preferably  $\text{H}_2\text{SO}_4$ , to prevent the precipitation of manganous oxid. Organic matter interferes and solutions cannot be filtered through paper; but asbestos, glass wool, or guncotton may be used.

The above solution is of such a strength that 17.35 c.c. will just oxidize one decigram of Fe.

The iron must be in the ferrous condition, and this condition may be brought about by the presence of magnesium or metallic zinc in  $\text{H}_2\text{SO}_4$  solutions, but all zinc or magnesium must be dissolved or removed before titration.

## Permanganate.

1 c.c. N/10 solution	.....	.0056	gram Fe;
1 c.c.    "        "	.....	.0072	"    FeO;
1 c.c.    "        "	.....	.008	"    Fe <sub>2</sub> O <sub>3</sub> .

The above factors will furnish data for calculating the results to the form indicated.

*Determination.*—Weigh out about one gram of metallic iron wire, dissolve in dilute  $H_2SO_4$  by aid of heat. Use a small flask closed with cork, carrying a small glass tube to act as reflex condenser. When dissolved, add 50 c.c. of recently boiled distilled water and titrate with permanganate, using no indicator.

Or, weigh out of ammonium ferrous sulfate  $(Fe(NH_4)_2(SO_4)_2 \cdot 6H_2O)$  .7 gram, dissolve in water, and add 5 c.c. of dilute  $H_2SO_4$  (1 to 5), and titrate with the permanganate until rose-color is permanent. .7 gram of above salt equals .1 gram Fe.

*Questions.*—In titrating with permanganate, why should free acid be present? Why is  $H_2SO_4$  preferred? Why cannot filter-paper be used in this work? Why must the Fe be in the ferrous condition? In what way is the ferric changed to the ferrous by the presence of zinc? In iron determination why use a flask with reflex condenser? Why use freshly boiled distilled water?

## 26. Calcium Determination.

Of calcium carbonate weigh out .5 gram, bring into solution, make up to 200 c.c., and use 50 c.c. for each determination. Precipitate as an oxalate, and dissolve ppt. in small quantity of hot HCl; wash into flask, make up to about 50 c.c. with fresh-boiled distilled water, acidify with  $\text{H}_2\text{SO}_4$ , warm to about  $75^\circ \text{C}$ ., and titrate with decinormal permanganate to a faint pink tinge. 1 c.c. permanganate = .0028 gram of CaO, or .0050 gram  $\text{CaCO}_3$ .

*Questions.*—Why is it necessary to precipitate the Ca as an oxalate? Why use  $\text{H}_2\text{SO}_4$  to acidify with? What is the reaction between the permanganate and the Ca?

## 27. Chlorin.

For determination of Cl volumetrically, a standard N/10 silver solution is necessary. To produce this, weigh out 17 grams of  $\text{AgNO}_3$  and make up to 1000 c.c. with distilled water and keep in dark. 1 c.c. of this solution is equivalent to .005837 gram NaCl or to .003537 gram Cl.

*Determination.*—Weigh out about one gram NaCl, make up to 200 c.c. with distilled water, and place exactly 20 c.c. in a white porcelain dish; add a few drops of saturated solution of potassium chromate, and titrate with N/10 silver solution. Each c.c. of

silver solution required in "titre" equals .003537 gram Cl or .005837 gram NaCl. A standard solution of sodium chlorid may be used for determining silver.

*Questions.*—What is the reaction between the silver solution and the Cl? What is the product formed at the end reaction? What other product is formed at the end reaction?

## CHAPTER IV.

### THE ANALYSIS OF ASHES AND SOILS.

#### 28. Ash Analysis.

THE material for ash analysis should be burned at low heat until well freed from carbon, then thoroughly pulverized in an agate mortar, and preserved in well-stoppered bottles until wanted.

*Solutions.*—Weigh out one gram of ash in a platinum evaporator, add a little water, then invert a small glass funnel in evaporating-dish, and add cautiously HCl until effervescence ceases; then a few drops of HCl in excess, and evaporate the whole to dryness on water-bath. Moisten the residue with HCl, add water, and heat on water-bath for a few minutes, then filter through a tared Gooch crucible. The residue is sand, silica, and carbon. Dried, weighed, and ignited, the loss is taken as carbon, while the residue is taken as sand and silica.

Dilute the filtrate to 200 c.c., and measure out 50 c.c. of the filtrate, 50 c.c. for each determination, or .25 grams of ash. Three portions are measured out in beakers A, B, and C.

**A. SULFURIC ACID.**—Dilute solution A to 100 c.c. or more, heat to boiling, and add barium chlorid to ppt.  $\text{BaSO}_4$ . Complete determination as in  $\text{H}_2\text{SO}_4$ .

*Alkalis.*—Heat the filtrate from above and add  $\text{Ba(OH)}_2$  in slight excess, then 1 c.c.  $\text{NH}_4\text{OH}$  and ammonium carbonate in slight excess, now add about .5 gram of dry powdered oxalic acid; filter, and wash well with hot water and evaporate filtrate to dryness, adding 5 c.c. of  $\text{HCl}$  to convert alkalis into chlorid. Determine Na and K as in method for separating these metals.

**B. IRON AND ALUMINA.**—To the 50 c.c. of original solution add  $\text{NH}_4\text{OH}$  to alkalinity, then a few drops of sodium acetate, and acetic acid in excess. The precipitate is ferric and aluminic phosphate. After drying, igniting in tared crucible, and weighing the desiccated product, the iron and alumina may be separated as follows: Bring residue of iron and alumina into a beaker, add 15 c.c. of dilute  $\text{H}_2\text{SO}_4$  ( $1 \text{ H}_2\text{SO}_4$  to 4  $\text{H}_2\text{O}$ ), heat on water-bath with beaker covered until in solution, then determine Fe with potassium permanganate by volumetric method, and from these results and total iron and aluminum phosphate estimate the amount of iron and alumina.

*Lime.*—Heat filtrate from last, add ammonium oxalate, allow to stand a few hours, and determine  $\text{CaO}$  as in calcium determination.

*Magnesia.*—Make filtrate from last alkaline with



$\text{NH}_4\text{OH}$  and add sodium hydrogen phosphate, and allow to stand for twelve hours; filter, wash with dilute ammonia, and weigh precipitate as  $\text{Mg}_2\text{P}_2\text{O}_7$  by methods already given.

C. PHOSPHORIC ACID.—To 50 c.c. of original solution add about 15 grams dry ammonium nitrate and 50 to 75 of molybdic solution, and continue the determination as for phosphate under apatite.

*Chlorin.*—Weigh out one gram ash, add water and a few drops  $\text{HNO}_3$ , heat on water-bath for a few minutes, filter and wash with hot water and determine Cl by means of standard  $\text{AgNO}_3$  solution, using potassium chromate as an indicator or the chlorin may be precipitated with silver and weighed gravimetrically.

*Carbon Dioxid.*—Weigh out 3 grams of ash and determine carbon dioxid same as in carbonic acid determination.

*Questions.*—Why moisten the residue from ash with  $\text{HCl}$  before dissolving and filtering? In determining the alkalis why add oxalic acid? In separating Fe and Al what reactions take place?

## 29. Soil Analysis.

The sample of soil for analysis should be well air-dried, and then passed through a sieve with circular openings one-half millimeter in diameter, using if necessary a rubber-tipped mortar to crush lumps, but

not to grind sample. The fine portion to be used for analysis should be preserved in a well-stoppered bottle.

*Moisture.*—Dry in water-oven on watch-glasses about 3 grams of soil until weight remains nearly constant. Loss is moisture.

*Volatile Matter.*—Heat the soil as dried above in a platinum dish to full redness until all organic matter is burned. Loss in weight represents volatile matter.

*Analysis Proper.*—In an Erlenmeyer flask of 200 c.c. capacity place 10 grams of air-dried soil and 100 c.c. of HCl, sp. gr. of 1.115. Close flask with cork, carrying a glass tube about 2 ft. in length to act as reflux condenser. Place flask in water-bath and keep at boiling temperature for ten hours with occasional shaking. Transfer contents of flask to beaker, and bring residue on to filter and wash with distilled water; dry residue and weigh as insoluble material. Make solutions and washings up to 500 c.c. with distilled water.

*Iron and Alumina.*—100 c.c. of above solution are made slightly alkaline with  $\text{NH}_4\text{OH}$ , then boiled to expel excess of ammonia, filtered, and well washed with hot distilled water. The precipitate is iron, alumina, and phosphates. These may be separated as in ash analysis.

*Manganese.*—The filtrate above is concentrated to about 100 c.c.,  $\text{NH}_4\text{OH}$  added to alkalinity, bromine water added and the solution heated to boiling.

Allow the solution to cool and add more bromin water and ammonia, and heat as before to precipitate all the manganese; acidify with acetic acid, and filter. While still boiling, wash with hot water and ignite as  $Mn_2O_4$ .

*Calcium*.—Evaporate above filtrate to about 50 c.c., add ammonia to slight alkalinity, and to the hot solution add ammonia oxalate, and determine CaO as by methods already given.

*Magnesia*.—If necessary concentrate the filtrate above, make alkaline with  $NH_4OH$ , and add  $Na_2HPO_4$ , and determine Mg as under magnesia determination.

*Alkalis*.—To the last filtrate add  $NH_4OH$ , boil to expel excess of ammonia, filter, and wash. Evaporate the filtrate to dryness, heat below redness to expel ammonia salts, add 30 c.c. of water, then a few drops of  $Ba(OH)_2$  solution, and heat to boiling. Filter, and wash with hot water. To the filtrate add  $NH_4OH$  and ammonium carbonate, filter, and wash with hot water. Evaporate to dryness and determine total alkalis, and make separation of K and Na as in method already given.

*Carbon Dioxid*.—In 5 grams of the air-dried soil determine carbon dioxid by method already described under carbonic acid determination.

*Chlorin*.—20 grams of soil are placed in flask and 100 c.c. water added, and with frequent shakings allowed to stand for two hours, then filtered, and the filtrate and washings made up to 200 c.c. In 50 c.c.

of solution determine Cl by volumetric method, with standard  $\text{AgNO}_3$  solution, using potassium chromate as an indicator. A porcelain dish is best for making the "titre" in.

### 30. Nitrogen Determination.

Of the air-dried soil weigh out 3 grams, place in digestion-flask, add 30 c.c. of concentrated sulfuric acid (sp. gr. 1.84) containing one gram of salicylic acid, and thoroughly mix by shaking. Heat over a low flame until no further frothing, and then boil briskly for five or ten minutes; now add about 0.7 gram of mercuric oxid, and continue the boiling until solution is colorless; then complete oxidation by adding slowly fine pulverized potassium permanganate to the hot solution, until after shaking the solution is green or purple in color. Transfer the contents of the cool flask to a distilling flask, using about 100 c.c. of water, and add a few pieces of granulated zinc to prevent bumping; now add 30 c.c. of potassium sulfid solution (40 grams  $\text{K}_2\text{S}$  in 1000 c.c.  $\text{H}_2\text{O}$ ), and after shaking add 80 c.c. of caustic soda solution or enough to make it alkalin (saturated solution of  $\text{NaOH}$ ), connect at once with the condenser, and distil over about 125 c.c., receiving into a measured quantity of standard acid, and then titrate with standard alkali, using cochineal as an indicator. 1 c.c. of the decinormal  $\text{H}_2\text{SO}_4$  is equal to .0014 gram of nitrogen.

In absence of nitrates the salicylic acid may be omitted from the  $\text{H}_2\text{SO}_4$ ; otherwise the nitrogen determination is made the same as above, with varying amount of substance to be analyzed, according to its nitrogen content.

*Questions.*—What is the action of the  $\text{H}_2\text{SO}_4$  on the organic matter? Why is salicylic acid necessary in the presence of nitrates? Why was  $\text{HgO}$  added? Why not add the  $\text{HgO}$  first? What is the action of the potassium permanganate? Before distilling, why add  $\text{K}_2\text{S}$ ? What change takes place on adding the  $\text{NaOH}$  solution? In what form did the  $\text{N}$  exist in the soil? In what manner was it all transformed into ammonia? Why use cochineal as the indicator?

## CHAPTER V.

### ANALYSIS OF ORES.

#### 31. Iron Ores.

*Moisture*.—Dry the well-pulverized ore on a watch-glass (2 to 3 grams) for two hours in water-bath.

*Carbon Dioxid*.—Introduce one gram of the pulverized ore into the  $\text{CO}_2$  apparatus, and determine by usual method.

#### MAIN ANALYSIS.

*Silica, etc.*—Place 10 grams of the finely pulverized ore in a porcelain dish, add concentrated  $\text{HCl}$ , and heat on water-bath for thirty minutes. Evaporate the solution to dryness. Add 10 c.c.  $\text{HCl}$ , warm on water-bath, add water, and after settling decant through filter into 500 c.c. flask; to the residue add  $\text{HCl}$ , heat, and add water, decanting as before, repeating if necessary until residue is free from iron; then bring the residue on to filter, wash with hot water, dry, and ignite as gangue and silica. The filtrate is then made up to the 500 c.c. mark.

*Sulfur*.—Evaporate 100 c.c. of filtrate nearly to

dryness, to expel acid; dilute with 150 c.c. of water, boil, and add a few drops of  $\text{BaCl}_2$  to ppt.  $\text{BaSO}_4$ ; allow to stand twelve hours, filter, and determine  $\text{SO}_3$  in precipitated  $\text{BaSO}_4$ .

*Phosphoric Acid.*—To 100 c.c. of the original filtrate after heating add about 15 grams  $\text{NH}_4\text{OH}$ , then the molybdate solution, and allow to stand in a warm place for twenty-four hours; filter, and wash with ammonium nitrate solution. The precipitate on filter-paper is then dissolved with  $\text{NH}_4\text{OH}$  and hot water, nearly neutralized with  $\text{HCl}$ , and “magnesia mixture” added to ppt.  $\text{P}_2\text{O}_5$ . After standing fifteen minutes add 30 c.c. of  $\text{NH}_4\text{OH}$ , and allow to stand for twenty-four hours. Complete determination in usual manner. If the ore is arsenic bearing, to this filtrate, before adding the molybdate solution, run in  $\text{H}_2\text{S}$  to precipitate the As as a sulfid. Heat the filtrate to expel  $\text{H}_2\text{S}$ , and proceed as above.

*Iron, Alumina, and Phosphates.*—100 c.c. of the original solution is boiled with a few drops of  $\text{HNO}_3$ , then acid ammonium carbonate added until the liquid is nearly neutral; then to the red solution add ammonium acetate in excess, boil for some time, and allow to settle. Filter (filtrate A), wash with hot water, dry, ignite, and weigh as  $\text{Fe}_2\text{O}_3$ ,  $\text{Al}_2\text{O}_3$  and phosphates. Deduct the weight of phosphates already determined, and residue is iron and alumina.

*Separation of Iron and Alumina.*—Place precipitate

in flask, add dilute  $\text{H}_2\text{SO}_4$ , and heat on water-bath until dissolved; make up to 200 c.c., measure out 50 c.c., and titrate with standard permanganate solution.

*Manganese.*—To filtrate A add a few drops of Br water, heat to  $50^\circ \text{C}$ ., tightly cork flasks, and allow to stand for twelve hours. The manganese will precipitate, and then may be filtered off, dried, ignited, and weighed as  $\text{Mn}_2\text{O}_3$ .

*Lime.*—Concentrate last filtrate to about 100 c.c., add  $\text{NH}_4\text{OH}$  and ammonium oxalate to precipitate Ca.

*Magnesia.*—Evaporate last filtrate to dryness, ignite at low redness, redissolve, and add one gram oxalic acid; evaporate to dryness and heat to low redness. Redissolve alkalis, filter, wash, dry, ignite, and weigh as  $\text{MgO}$ .

*Alkalis.*—Evaporate filtrate from above to dryness, after adding a few drops of  $\text{HCl}$ , ignite, and weigh as total alkali. The potash and soda may then be separated by usual methods, if desired.

*Questions.*—Why was  $\text{HCl}$  added to residue before redissolving for the silica? In what form was S in the ore? How was it converted to the sulfate? In  $\text{P}_2\text{O}_5$  determination, why add  $\text{NH}_4\text{OH}$  before adding the molybdate solution? Why not wash precipitate with water? Why was ammonium acetate used in precipitating iron? Why was  $\text{HNO}_3$  added? What is the action of the Br in the Mn determination?



### 32. Zinc Blende.

Place about one gram of the finely pulverized ore in a porcelain dish and add  $\text{HNO}_3$ . This dish should be covered and gradually heated on water-bath. When all action has ceased, evaporate to dryness, moisten with  $\text{HCl}$ , add water, heat, and then filter and wash precipitate with hot water. The residue is gangue. To the filtrate add  $\text{H}_2\text{S}$  to saturation, and then filter. Boil filtrate to expel all of the  $\text{H}_2\text{S}$ . Add a few drops of  $\text{Br}$  water to oxidize any iron, add  $\text{NH}_4\text{OH}$  in slight excess, then a few drops of potassium permanganate, and boil and filter to remove iron. To the filtrate while hot add acetic acid to acidulate, and then  $\text{H}_2\text{S}$  until the zinc is all precipitated. Wash by decantation with hot water containing  $\text{H}_2\text{S}$ , filter (filtrate B), and dissolve precipitate in  $\text{HCl}$ . Add water and a few crystals of  $\text{KClO}_3$ , and boil; filter off  $\text{S}$ , and to filtrate add  $\text{Na}_2\text{CO}_3$  to precipitate zinc as a carbonate, filter, wash with hot water, dry, and ignite in a porcelain crucible as  $\text{ZnO}$ .

Filtrate B from the  $\text{ZnS}$  is boiled, and ammonium oxalate added to precipitate  $\text{CaO}$ .

To the last filtrate add  $\text{NH}_4\text{OH}$  and then sodium acid phosphate to precipitate  $\text{Mg}$ , which is weighed as  $\text{Mg}_2\text{P}_2\text{O}_7$ .

Instead of dissolving zinc in  $\text{HCl}$ , nitric acid may be used, the excess of  $\text{HNO}_3$  evaporated off and

microcosmic salt added in excess, the solution heated to boiling,  $\text{NH}_4\text{OH}$  added to alkalinity, and the precipitate ignited and weighed as  $\text{Zn}_3\text{P}_2\text{O}_7$ . Or, the sulfid may be treated in the crucible with a few drops of  $\text{HNO}_3$  and  $\text{H}_2\text{SO}_4$ , and then gently heated on a plate some distance above flame, until the sulfid is transformed into a sulfate, and as such weighed.

*Questions.*—Why use  $\text{HNO}_3$  instead of  $\text{HCl}$  for dissolving the blende? Why add  $\text{H}_2\text{S}$  before precipitating  $\text{Zn}$ ? Why use  $\text{H}_2\text{S}$  in wash-water? Why ignite in porcelain?

### 33. Galena.

Of the finely pulverized ore, weigh out about one gram, add 20 c.c. of concentrated  $\text{HNO}_3$  in covered porcelain dish, and heat slowly on water-bath. Evaporate nearly to dryness, then add 8 c.c. of water and heat on water-bath; then add 5 c.c. concentrated  $\text{H}_2\text{SO}_4$ , and continue heating until  $\text{SO}_3$  fumes are given off; then allow to cool, and add 100 c.c. water and heat to boiling, breaking up the precipitate by stirring. Allow to cool and decant, then wash the precipitate on to filter with a 1 per cent  $\text{H}_2\text{SO}_4$  solution, and finally wash with alcohol. Transfer precipitate to porcelain dish, add a hot concentrated solution of ammonium acetate, made slightly acid with acetic acid, to dissolve the  $\text{PbSO}_4$ . Filter into a beaker, pouring the filtrate through the filter several times;

then wash with hot water. To the filtrate add  $\text{H}_2\text{SO}_4$ , allow to stand until precipitate has settled, then filter, wash with 1 per cent  $\text{H}_2\text{SO}_4$ , and finally with alcohol; dry, ignite filtrate separately, oxidizing with  $\text{HNO}_3$ , and then add the ash to the precipitate and ignite as for lead.

To the filtrate, after adding 100 c.c. water, add a few drops of  $\text{HCl}$  to precipitate  $\text{Ag}$  if present; filter, and determine  $\text{Ag}$  as  $\text{AgCl}$ .

To the filtrate add  $\text{NH}_4\text{OH}$  to alkalinity to precipitate  $\text{Fe}$ , and determine as for  $\text{Fe}$  determination.

To filtrate add  $\text{H}_2\text{S}$  to precipitate the  $\text{Cu}$ ,  $\text{Zn}$ , etc., as sulfids, which may then be separated and determined by the methods already given.

*Questions.*—Why redissolve precipitate with ammonium acetate? Why reprecipitate with  $\text{H}_2\text{SO}_4$ ? Why add  $\text{H}_2\text{SO}_4$  to the wash-water?

### 34. Coal Analysis.

*Moisture.*—Place one to two grams of the finely pulverized coal in a platinum crucible, and heat in an air-bath at  $110^\circ$  for fifteen minutes. Cool and weigh. Repeat for fifteen minutes more, cool, and weigh to see if weight remains constant. Loss is moisture.

*Volatile Matter.*—Heat the above crucible with contents for about three and one-half minutes over Bunsen burner, and then for three and one-half minutes over blast-lamp. The crucible should remain

covered during the entire process. Cool and weigh. Loss is volatile matter, and one-half of the sulfur that may be contained in the coal in form of sulfid.

*Fixed Carbon.*—Heat the above crucible until ash is white. Cool, and weigh, the loss being fixed carbon and one-half of any sulfur that may have been in the coal.

*Sulfur.*—Place 1.5 grams of the finely pulverized coal in flask, and add concentrated  $\text{HNO}_3$  and potassium chlorate to oxidize all the organic matter; filter, and to the hot filtrate add a little  $\text{HCl}$ , then  $\text{BaCl}_2$  in slight excess; allow to settle, filter, wash, dry, ignite, and weigh as for sulfuric acid determination. The weight of the precipitate multiplied by  $16/116.5$  gives the weight of sulfur in the coal.

## CHAPTER VI.

### ELECTROLYSIS.

ELECTROLYSIS is the galvanic decomposition of compounds, called electrolytes. Electrolytic analysis is the decomposition of a compound, and depositing of the radicle or element in a form to be weighed or "titred." Water acidulated with  $H_2SO_4$  and a galvanic current passed through it is decomposed into its elements H and O. One of the elements or groups of a compound separates at the positive pole or anode and is called anion, and the other separating at the negative electrode is called cation or electropositive constituent or element. The metals are electropositive or cations, while the non-metals are electronegative or anions, and appear at the positive pole.

The ohm is the unit of resistance, and is equivalent to the resistance offered by a column of pure mercury 106.2 centimeters in length and one square millimeter in section at a temperature of  $0^\circ C$ .

The volt is the unit of electromotive force, and produces a current of one ampere with a resistance of one ohm.

Ampere is a unit of current, and is produced by an electromotive force of one volt acting against a resistance of one ohm.

*Batteries.*—The Bichromate, Leclanché, Grover, Daniell, Crowfoot, or Smee battery may be employed. I have found the Crowfoot and Smee batteries to be satisfactory for student use, for such work as has been undertaken. Expensive apparatus is not necessary, and laboratory improvised pieces have been used by the students, for the work mentioned beyond, as successfully as the most expensive apparatus. Only enough work in electrolysis is undertaken to give the student a knowledge of the processes, and those desiring further work along this line are referred to the special works devoted to this important subject.

*Arrangement of Apparatus.*—A clean platinum dish free from dents—preferably one used for no other purpose—is employed to hold the solution to be electrolyzed, and upon the inner surface of which the metal is to be deposited. To the arm of an ordinary filter-stand is attached a binding-screw, to which is attached the positive pole or wire from the battery and the spiral coil of platinum wire that dips into the solution to be electrolyzed. On the base of the filter-stand is placed a piece of copper plate, upon which the platinum dish rests, and a binding-screw through the copper plate or ring serves to connect the negative

wire or pole from the batteries. From one to three batteries are used for the decomposition of copper, iron, or nickel. If a voltmeter is available, the exact current desired can be secured for each particular determination; thus for electrolyzing Cu, a current giving from 0.3 to 0.5 c.c. of electrolytic gas per minute is all that is necessary from the sulfate solution, or 3 to 4 c.c. from ammonium oxalate solutions. For nickel, a current of 5 c.c. electrolytic gas is best, and for iron 8 to 10 c.c. per minute.

An ampere is equal to 10.436 c.c. of electrolytic gas per minute. By electrolytic gas is meant oxygen-hydrogen gas decomposed from acidulated water by the current employed.

### 35. Copper.

Dissolve 4.9 grams of the pure copper sulfate in water and make up to 200 c.c. Place 50 c.c. of this solution in a platinum dish, the weight of which is accurately known. The negative pole of the battery is then connected with the dish, and the positive pole is connected with a coiled spiral of platinum wire that just dips into the solution, but does not touch the dish. Continue the process until all of the Cu is deposited and no reaction is shown with a freshly prepared solution of potassium ferrocyanid. For the successful precipitation of Cu a weak current is neces-

sary. A strong current deposits the Cu in a spongy condition. The deposited copper should then be washed carefully with hot distilled water, then with alcohol, dried, and weighed. (The 50 c.c. above contain exactly .25 gram of Cu.)

### 36. Nickel.

Dissolve 3.586 grams of pure nickel sulfate in distilled water and make up to 200 c.c. Place 50 c.c. of the solution in a platinum dish carefully weighed, and add  $\text{NH}_4\text{OH}$  to strong alkalinity. Place the dish in connection with the negative pole, and the positive pole attached to a platinum electrode is placed in the solution, and the process continued until the Ni is all precipitated; then wash with hot water, followed by alcohol; dry at  $110^\circ$ , cool, and weigh. (The 50 c.c. of solution contain exactly .20 gram of Ni.) A strong current is required for depositing Ni. Two and perhaps three Bunsen cells will be required to do the work in twelve hours.

### 37. Iron.

Place 1.5 grams of pure iron sulfate in flask, add water to dissolve, then a saturated solution of ammonium oxalate in excess until precipitate redissolves and solution is yellowish red in color; then make up to 200 c.c. Place 50 c.c. of this solution in a tared



platinum dish, add 50 c.c. of water, and electrolyze as for nickel. While electrolyzing the solution, for good results the solution should be kept at 65° to 70° C., when the iron will be deposited as a steel-gray product. The electrolyzed solution should give no reaction for iron when a few drops are made acid with HCl and potassium sulfocyanid is added. Should  $\text{Fe}(\text{OH})_3$  be deposited during the process, this precipitate may be again redissolved by adding drop by drop oxalic acid. A strong current is required for depositing the iron.

## CHAPTER VII.

### SUGARS, STARCHES, FOODS AND MILK.

#### 38. Fehling's Solution.

1. 69.278 grams of crystallized copper sulfate are dissolved in water and made up to 1000 c.c.

2. 346 grams of Rochelle salts and 80 grams of NaOH are dissolved in water up to one litre. These two solutions are kept separate and mixed together in equal volumes when wanted for use.

3. 104 grams of iron ammonium alum are weighed out, dissolved in water, 40 c.c. of  $H_2SO_4$  added, and the solution made up to 1000 c.c.

4. 3.5 grams of potassium permanganate are weighed out, placed in a flask, and diluted to 1000 c.c. with distilled water.

Solution 4 should be standardized by using cane-sugar of known purity. One c.c. of the permanganate is equivalent to 6.9 mg. of copper reduced to cuprous oxid. Or, for convenience in calculations, the following table is inserted showing milligrams of sucrose and of invert-sugar for each c.c. of permanganate from

1 c.c. to 64 c.c. The other sugars and starch are readily calculated from the data given, for fractions of a c.c. of permanganate interpolations can easily be made.

TABLE SHOWING MILLIGRAMS SUGAR PER C. C.  
PERMANGANATE.

Permanganate, C. C.	Sucrose, Milligrams.	Invert Sugar, Milligrams.	Permanganate, C. C.	Sucrose, Milligrams.	Invert Sugar, Milligrams.
1	3.0	3.17	33	115.0	121.05
2	6.1	6.42	34	118.8	125.05
3	9.2	9.67	35	122.5	128.94
4	12.3	12.93	36	126.3	132.94
5	15.5	16.31	37	130.0	136.84
6	18.8	19.79	38	133.8	140.84
7	22.0	23.15	39	137.6	144.84
8	25.2	26.52	40	141.5	148.94
9	28.5	30.00	41	145.4	153.04
10	31.8	33.47	42	149.3	157.15
11	35.1	36.94	43	153.2	161.26
12	38.5	40.52	44	157.2	165.47
13	42.0	44.21	45	161.1	169.57
14	45.4	47.79	46	165.0	173.68
15	48.9	51.47	47	169.0	177.89
16	52.4	55.15	48	173.0	182.10
17	56.0	58.94	49	177.1	186.42
18	59.6	62.73	50	181.1	190.63
19	63.2	66.47	51	185.2	194.94
20	66.9	70.42	52	189.5	199.47
21	70.4	74.10	53	193.5	203.68
22	74.1	78.00	54	197.6	208.00
23	77.9	82.00	55	201.8	212.42
24	81.4	85.68	56	206.0	216.84
25	85.0	89.47	57	210.2	221.27
26	88.9	93.57	58	214.6	225.89
27	92.5	97.37	59	219.0	230.52
28	96.3	101.37	60	223.5	235.26
29	100.0	105.26	61	227.9	239.89
30	103.8	109.26	62	232.2	244.42
31	107.5	112.10	63	236.6	249.05
32	111.2	117.05	64	241.0	253.68

### 39. Methods of Determination.

*Sugar.*—The solution to be tested should not contain more than 2 per cent of sugar; solutions containing more than this should be diluted to a known volume and an aliquot part taken.

*Invert sugar.*—Place 10 c.c. of the sugar solution in a flask or beaker, add 50 c.c. of Fehling's solution (Nos. 1 and 2 in equal volume), and bring to a boil; continue the heating on water-bath for ten minutes, filter through an asbestos filter, wash with hot water, transfer filter and contents again to flask, add 100 c.c. of the iron-ammonium-alum solution, and titrate at once with permanganate.

*Sucrose.*—A known quantity of the solution is treated with concentrated HCl, then heated on water-bath for 30 minutes, neutralized with  $\text{Na}_2\text{CO}_3$ , made up to a known volume, and sugar determined in 10 c.c. as for invert-sugar. From total amount deduct amount of invert-sugar above and balance is sucrose.

### 40. Food Analysis.

The air-dried substance should be ground fine and kept in well-stoppered bottles for use.

*Moisture.*—About 2.5 grams of substance may be dried for eight hours in water-oven on watch-glass, or better in current of hydrogen when possible. The loss in weight is moisture.

*Ether Extracts.*—A convenient piece of apparatus

may be made by perforating a small hole in bottom of test-tube, either by grinding on stone or by heating in flame and blowing out. A small wad of recently-ignited asbestos is put in the bottom and a little asbestos pulp added, the filter is then washed and dried, and the food used for moisture determinations transferred to this. Into the tube, upon the food, drop a small piece of glass rod, and place the whole in a Caldwell extraction-tube, attach to a condenser, and attach a small weighed flask to the bottom to receive the extract; ether is now added and the substance is allowed to extract continuously for eight hours. The flask beneath the extractor is kept nearly submerged in boiling water during the process of extraction. The ether is then driven off, the flask dried in the water-oven, and when cooled weighed, at once giving the amount of ether extract, often taken as fat.

*Ash.*—About 2 to 3 grams of the substance is weighed into a platinum crucible and charred at lowest possible heat, and then the burning continued at low red heat until ash is white.

*Nitrogen.*—Place in digestion-flask .5 gram of the air-dried substance and treat as for nitrogen in soil analysis, omitting the salicylic acid from  $H_2SO_4$ , and 15 c.c. of the latter will usually be enough, instead of 30 c.c. as in soil analysis.

*Crude Protein.*—The nitrogen multiplied by 6.25 will give the crude protein.

*Albuminoid Nitrogen. Reagent.* — Dissolve 100 grams of pure copper sulfate in 5000 c.c. of water, add 25 c.c. of glycerol, and then gradually a dilute solution of NaOH until alkalin; filter, rub the ppt. up with water containing 5 c.c. of glycerol per 1000 c.c., filter, and wash the precipitate until the water is no longer alkalin. Rub the ppt. up with a 10 per cent glycerol solution until it will flow well from a pipette. Determine the  $\text{Cu}(\text{OH})_2$  per cubic centimeter of this solution. Weigh out .7 gram of air-dry substance, add 100 c.c. of water, heat to boiling in a beaker on water-bath and add enough of the cupric hydrate solution to equal about .5 gram  $\text{Cu}(\text{OH})_2$ ; filter, and wash with cold water, dry precipitate, transfer to nitrogen flask, and determine nitrogen as in above case, but use nearly double the amount of  $\text{K}_2\text{S}$ . The per cent of  $\text{N} \times 6.25 =$  albuminoids.

*Carbohydrates.* — Of late much attention has been given to a study of the carbohydrates, and experimentally the method of Stone is given for this work.

*Invert-sugar.* — Weigh out of the material 10 grams, place in flask, add 100 c.c. 95 per cent alcohol, and boil with a reflux condenser on water-bath for two hours; filter, and determine sugar in aliquot part by method already given, first evaporating off alcohol and taking up sugar in water.

*Sucrose.* — A portion of the above solution is evaporated to dry off alcohol and the sugar taken up with

water, the sucrose inverted by means of HCl, and sugar determined by means of Fehling's solution, as in last. Correcting for invert-sugar present, we have sucrose or cane-sugar.

*Dextrin.*—The residue from the alcohol extraction is returned to flask, 500 c.c. of water added, and the whole allowed to stand for twenty-four hours with frequent agitation. Filter through fine linen and concentrate on water-bath to 200 c.c. In an aliquot part of this solution invert with HCl, and determine total invert-sugar by means of Fehling's solution. In another portion precipitate the soluble starch by adding barium hydroxid solution in excess, filter, and invert by means of HCl, and determine invert-sugar, which is taken as the amount of dextrin.

*Soluble Starch.*—The total invert-sugar, above, less that as dextrin, gives the soluble starch.

*Starch.*—The residue from the aqueous extract is returned to the flask and 100 c.c. of water added, the flask closed with a reflux condenser, and the contents boiled for two minutes to convert the starch into a soluble form. To the solution cooled to 60° 10 c.c. of malt\* infusion is added and allowed to act at a temperature of not to exceed 65° from two to three

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\* To prepare malt infusion 10 grams of fine-ground fresh malt are covered with 50 c.c. of water and kept at temperature of work room from two to three hours with frequent shaking. The solution is then filtered and is ready for use.

hours, or until there is no blue starch reaction with iodine. The solution is then filtered, using a fine linen filter, washed with hot water, and the filtrate concentrated on water-bath to 100 c.c. Transfer the filtrate to a 200-c.c. flask, and add 10 c.c. of concentrated HCl and keep at temperature of boiling water on bath for one hour to convert all the sugar into dextrose. The solution is then neutralized with  $\text{Na}_2\text{CO}_3$ , and sugar determined with Fehling's solution. A determination must be made of the sugar contained in the malt extract and a correction made for the same. The starch may then be calculated from the inverted sugar.

*Pentosans.*—Remove carefully from the linen filter the residue from the starch determination, place in a flask with 100 c.c. of water, add 2 c.c. of concentrated HCl and keep at boiling temperature for one hour, using reflux condenser to prevent evaporation. By this treatment the most of the gums, pentosans, etc., are converted into reducing-sugars, taken as xylose. The solution is filtered, washed, made up to 200 c.c., and the sugar determined by means of Fehling's solution.

*Crude Fiber.*—The residue from the above is brought into a flask and 200 c.c. of 1.25 per cent NaOH solution added, boiled for 30 minutes, filtered on an asbestos filter, washed with hot water, and finally with ether; then dried, transferred to a platinum dish,



weighed, ignited, and again weighed, the loss being taken as crude fiber.

*Undetermined Carbohydrates.*—The sum of all products thus far determined from 100 gives the amount of undetermined carbohydrates.

*Questions.*—Is the ether extract pure fat? What else does it contain? About what proportion of the ether extract is pure fat?

In what form did the nitrogen exist in the plant? How is it converted into ammonia? Why multiply the per cent of N by 6.25?

What is the active principle in the malt infusion? What is the nature of its action on the starches? Are the starches of all foods alike?

What is the fiber of the food?

#### 41. Milk Analysis.

Great care should be taken to procure a uniform sample of the product, and before taking each portion for analysis the sample should be well mixed by pouring several times from one beaker to another.

*Ash.*—A small platinum evaporating-dish containing a quantity of asbestos should be ignited, cooled, and weighed. Place in this about 5 c.c. of the milk, weigh, place in water-oven to dry, and then burn at low redness. Cool, and weigh as ash.

*Total Solids.*—In a perforated test-tube, as used

for ether extract in food analysis, place a small wad of fat free cotton; fill tube loosely three-fourths full of freshly ignited pure asbestos, and have a small wad of cotton for top; the desiccated tubes should now be weighed. Then remove the wad of cotton from top, and run in upon asbestos about 3 c.c. of the well-mixed sample of milk; return the wad of cotton, and weigh. These tubes should then be placed in a water-oven and the perforated end of the tube connected with a suction-pump and rapidly dried. For accurate work, dry hydrogen gas instead of air should be drawn through the tubes while drying. Cool the tubes in a desiccator, and weigh to find total solids. The loss is water.

*Fat.*—The tubes containing the desiccated milk above may be placed in extractor and fat estimated in the same manner as ether extracts are found in food.

*Nitrogen.*—Weigh out 4 to 5 grams of milk in a flask and determine nitrogen in same manner as for foods. The milk need not be evaporated before adding the  $\text{H}_2\text{SO}_4$ . The percentage of nitrogen multiplied by 6.25 gives total nitrogen compounds, usually stated as casein. Many use the factor 6.37 in preference to 6.25.

*Milk-sugar or Lactose.*—20 grams of milk are placed in a porcelain dish with 30 c.c. of water, a few drops of acetic acid added and heated to 50 c.c. to coagulate the casein. The solution is then filtered through

fine linen, washed, and made up to 150 c.c., and containing a few drops of HCl heated on water-bath for thirty minutes to invert-sugar; then neutralized with  $\text{Na}_2\text{CO}_3$  made up to 200 c.c., and the sugar determined in 10 c.c. by means of Fehling's solution.

*Specific Gravity.*—A uniform sample of the milk should be brought to a temperature of 60° F., placed in a specific-gravity tube, and the hydrometer-reading taken directly from the scale.

*Questions.*—Is there any other nitrogenous constituent besides casein present in the milk? Is the reducing power of milk-sugar the same as that of cane-sugar?

## CHAPTER VIII.

### . 42. Water Analysis.

*Total Solids.*—100 c.c. of water is placed in a tared platinum dish and evaporated on water-bath, and then dried in water-oven. This gives total solids. Ignite at low heat, and loss is volatile matter. The results should be expressed as parts per million.

#### CHLORIN.

*Solutions.*—4.789 grams of  $\text{AgNO}_3$  are dissolved to 1000 c.c. with distilled water. One c.c. of this standard  $\text{AgNO}_3$  solution is equivalent to .001 gram of chlorin.

*Determination.*—100 c.c. of water are placed in a porcelain dish, a few drops of saturated solution of potassium chromate added, and the standard  $\text{AgNO}_3$  solution run in until permanent color is secured. The results should be expressed in parts per million.

#### FREE AMMONIA.

*Solutions.*—1. A standard solution of ammonium chlorid is prepared by dissolving 3.15 grams of am-

monium chlorid in pure distilled water and making up to 1000 c.c. One c.c. of this solution diluted to 100 c.c. will contain .00001 gram of ammonia per c.c.

2. Alkaline potassium permanganate is best made by dissolving 200 grams of potassium hydroxid and 8 grams of crystallized dry potassium permanganate in water, and making up to 1000 c.c. with ammonia-free water. This solution should give no reaction for ammonia. Should there be any ammonia a correction should be made for each determination.

3. The Standard Nessler Reagent may be made by dissolving 13 grams of pulverized mercuric chlorid (corrosive sublimate) in 500 c.c. of distilled water, and 35 grams of potassium iodid in 200 c.c. of water. Pour the mercuric chlorid solution into the potassium iodid and add gradually, drop by drop, a saturated solution of corrosive sublimate until a permanent red precipitate just begins to remain undissolved. To this solution add 120 grams sodium hydroxid, dissolved in 150 c.c. of water, and dilute the solution to 1000 c.c. Sensitize this solution by adding a little more of the saturated solution of mercuric chlorid until a slight permanent red precipitate forms; allow to settle, and decant. Keep the solution in a cool place, away from the light.

4. Sodium carbonate is prepared by boiling an excess of sodium carbonate in distilled water and decanting off solution after cooling.

5. Ammonia-free water is best prepared by redistilling distilled water, to which a little  $\text{H}_2\text{SO}_4$  has been added, rejecting the first portion and not distilling too low.

*Determination.*—A glass retort or distilling apparatus is thoroughly cleaned out and 500 c.c. of the water to be tested is placed in the retort and 1 to 2 c.c. of the solution No. 4 ( $\text{Na}_2\text{CO}_3$ ) is added, and four portions of 50 c.c. each are distilled over, being received in Nesslerizing tubes. Into each of the above tubes are run 2 c.c. of the standard Nessler reagent, and the whole well mixed by pouring from one tube to another.

In a second Nessler's tube, well cleaned with acid, ammonia-free water is placed containing a measured volume of the standard ammonia solution, No. 1, and then filled to the 50-c.c. mark with ammonia-free water and 2 c.c. of Nessler solution added. If the shade produced does not correspond in color with that of the water sample under examination, test solutions of known strength must be made until the desired shade is secured; from the data may be calculated the parts per million of free ammonia. Many chemists Nesslerize the first 50 c.c. only of the distillate and add one-third for the other three portions, which are thrown away. If one has a calorimeter, the work of Nesslerizing is much simplified.

## ALBUMINOID AMMONIA.

To the retort from which the free ammonia has been distilled 50 c.c. of the alkalin potassium permanganate (solution No. 2) are added, and the distillation continued until three portions of 50 c.c. each have been collected. These are to be Nesslerized as for free ammonia, and expressed as parts per million for albuminoid ammonia.

## NITRATES.

The solutions required for this work are as follows:

1. *Standard Potassium Nitrate.*—Dissolve .722 gram of pure potassium nitrate in 1000 c.c. of water. This solution is of such strength that 1 c.c. is equivalent to .00044 gram of  $\text{NO}_3$ .

2. *Phenol Sulfuric Acid.*—To 370 c.c. of sulfuric acid, sp. gr. 1.84, add 80 grams of chemically pure phenol and 30 c.c. of water.

*Determinations.*—Evaporate to dryness in porcelain dish on water-bath 25 c.c. of the water to be tested. Mix thoroughly with the residue 1 c.c. of the phenol sulfuric acid. To the residue add 1 c.c. of distilled water, three drops of  $\text{H}_2\text{SO}_4$ , sp. gr. 1.84, and warm on water-bath; dilute with 25 c.c. of distilled water, make alkalin with  $\text{NH}_4\text{OH}$ , and dilute solution to 100 c.c. with distilled water. If nitrates are present a yellow color is produced, the depth of color depend-

ing upon the amount of nitrate present, ammonium picrate being formed.

The determination is made by comparison with standard solution. Evaporate to dryness 1 c.c. of the standard potassium nitrate solution No. 1, and treat the residue in same manner as water. If a calorimeter is available the comparison of the known and the unknown solution can be made directly. If not as in free and albuminoid ammonia determination, other test solutions of differing strength will need to be made until the approximate shade is reached.

These solutions should be examined in Nessler tubes over white tile and surrounded by dark paper.

#### HARDNESS.

1. *Standard Hard Water.*—Dissolve one gram of pure calcium carbonate in dilute HCl, and evaporate to dryness. Dissolve in water and make up to 1000 c.c. One c.c. of this solution is equivalent to .001 gram of  $\text{CaCO}_3$ .

2. *Standard Soap Solution.*—Weigh out of pure Castile soap 10 grams and dissolve in 35 per cent alcohol and make up alcoholic solution to 1000 c.c. One c.c. of this soap solution will just neutralize .001 gram of carbonate of lime. As the soap is not always of the proper strength, it is necessary to standardize the solution by means of the standard



water as follows: A measured quantity of the hard water (No. 1), say 10 c.c., is made up to 100 c.c. with distilled water, and titrated with the soap solution. This is best done in a glass-stoppered bottle of about 200 c.c. capacity, and to the test solution the soap solution is added, 1 c.c. at a time, from a burette, then shaken, and this continued until a permanent lather persists for fully five minutes. As 1 c.c. of the soap solution is taken up by the distilled water the 10 c.c. above should require 11 c.c. of the standard soap solution. After a few trials, if the strength is not correct, the standard soap solution must be adjusted by dilution or addition of more soap until the correct strength is secured. The hardness of the water is determined in exactly the same manner as for standardizing above, using 100 c.c. of water and expressing the results in parts per million. The above determination gives the total hardness. It is frequently desired to determine the permanent and temporary hardness of the water which together constitutes the total hardness. If so, a second portion of the water under examination, say 250 c.c., is placed in a flask and boiled for thirty minutes, replacing the loss from time to time with hot distilled water. After cooling, the water is made up again with distilled water to 250 c.c., and the hardness determined in 100 c.c. of this. The result shows the permanent hardness; this, taken

from amount of total hardness, will show amount due to temporary hardness.

*Questions.*—In Cl determination, why was potassium chromate used? What was the change indicating the end reactions?

In ammonia determination, why add  $\text{Na}_2\text{CO}_3$  before distilling? What is the difference between the free ammonia and albuminoid ammonia in the water?

What do the presence of nitrates indicate? What is the composition of the nitrates as determined? How were they transformed to this end product? What is the difference between the permanent and temporary hardness of water? What reactions take place between the soap and the products in the water?

## CHAPTER IX.

### 43. Urine Analysis.

1. Note the time of day for taking sample, and if convenient the total amount voided in twenty-four hours should be saved for examination.

2. *Color*.—Normal urine from pale yellow to brownish.

3. *Transparency*.—Normal urine when freshly voided is clear, but shows on standing a slight cloudiness.

4. *Consistency*.—Normal urine is perfectly fluid.

5. *Odor*.—Fresh normal urine has a peculiar aromatic odor. If concentrated it is strong, and in case of ammoniacal decomposition it is strong, and may be even putrid.

6. *Reaction*.—When freshly voided, normal urine is usually slightly acid, generally increasing in acidity for a time, and then becomes alkaline.

7. *Specific Gravity*.—This is taken by means of a urinometer at a temperature of 15.5° C., and for normal urine may be expected to range from 1.015 to 1.025.

## NORMAL CONSTITUENTS.

*Urea.*

*Volumetric Determination.*—This is quite simple and accurate when conducted with proper care, and depends upon measuring the liberated nitrogen in a properly constructed apparatus. The urinometer furnished by Eimer & Amend, New York, is very satisfactory, and is so graduated as to read the grams of urea direct.

*Solution 1.*—In 250 c.c. of distilled water, dissolve 100 grams of NaOH.

2. *Bromin.*—This is unpleasant to handle, but by means of a small pipette with a rubber nipple it can be done with little inconvenience and no danger to the person.

*Process.* — Dilute solution No. 1 with an equal volume of water when ready for use, and fill the long arm and bulb of the urinometer with this solution, to which has been added 1 c.c. of bromin (No. 2). Of the well-mixed sample of urine draw up 1 c.c. into a small pipette with a curved point by aid of the rubber nipple, insert the end of the pipette well into the neck of the urinometer, and gradually force out the urine by compressing the nipple. Be careful not to force in any air from the pipette after the urine is expelled. Allow the apparatus to stand for a time

and make the reading at a temperature of  $15.5^{\circ}$  C. or  $60^{\circ}$  F.

### *Uric Acid.*

On standing, crystals of uric acid may form on sides and bottom of beaker. Urine containing large quantities of urates, on the addition of concentrated  $\text{HNO}_3$ , form a ring resembling albumen. On standing or by heating the ring disappears.

*The Murexid Test.*—On evaporating to dryness, with a few drops of  $\text{HNO}_3$ , urine supposed to contain urates, and then adding to the residue a few drops of ammonia water, there will be formed a beautiful purple or violet-red color.

*Approximate Estimation.*—To 300 c.c. of urine add 10 c.c. of  $\text{HCl}$ , and allow to stand in a cool place for twenty-four hours and the crystals of uric acid will form. Filter and wash; remove from the filter, dry, and weigh. By this method an approximate idea may be secured of the amount of urates present.

### *Indican.*

*Jaffe's Test.*—Place in test-tube 10 c.c. of clear urine, add 10 c.c. of concentrated hydrochloric acid, and mix well. To this is added, drop by drop, a freshly prepared concentrated solution of calcium hypophosphite until a blue color is produced, provid-

ing indican be present. Now add 3 c.c. of chloroform and shake well, and the color is wholly taken up by the chloroform. In place of the hypophosphite a concentrated solution of chlorid of lime will answer. An excess of the reagent should be avoided.

### *Chlorids.*

1. Dissolve 7.269 grams of pure  $\text{AgNO}_3$  in distilled water and make up to 250 c.c. One c.c. of this solution is equivalent to .01 gram  $\text{NaCl}$ .

2. Prepare a small quantity of a saturated solution of potassium chromate.

*Method.*—Place 10 c.c. of the urine in a flask and make up to 100 c.c. with distilled water. Place in a porcelain dish, add a few drops of  $\text{HNO}_3$  to prevent precipitation of phosphates, put in a few drops of potassium chromate solution (No. 2), and add with constant stirring the  $\text{AgNO}_3$  solution until the beginning of a permanent orange tint indicates the end reaction.

### *Phosphates.*

The phosphates are of two classes, alka<sup>li</sup>n and the earthy phosphates. Phosphates may be precipitated by heat, which precipitate must not be mistaken for albumen. The addition of a little acid to the urine will redissolve the phosphates or prevent their being formed,

*Alkaline Phosphates.*—To the urine add a few drops of acetic acid, and heat to precipitate any albumen; then filter, and add  $\text{NH}_4\text{OH}$  or  $\text{KOH}$ . Precipitate is alkaline phosphates.

*Earthy Phosphates.* — To the filtrate above add “magnesia mixture,” and the precipitate is due to earthy phosphates. In normal urine the alkaline phosphates constitute about two-thirds and the earthy phosphates one-third of the total amount.

#### ABNORMAL CONSTITUENTS.

*Albumen.*—Fill a test-tube two-thirds full of urine and add a few drops of acetic acid to prevent phosphates from being precipitated. Hold the tube obliquely over the flame so as to heat the upper portion to boiling. If precipitate is formed, add slowly, drop by drop, 10 to 20 drops of concentrated  $\text{HNO}_3$ ; if a cloudiness appears or is not redissolved by the nitric acid, then albumen is present.

Another test is to place in a test-tube about two inches of the urine to be examined, and then by means of a pipette, held in the urine, to allow concentrated  $\text{HNO}_3$  to flow down the side of the tube below the urine so as not to mix with it. If albumen be present at the juncture of the two liquids, there will be a cloudy ring. A similar ring higher up due to the presence of urea or uric acid must not be mistaken for albumen.

*Sugar.*—Heat the urine to be examined and filter, and then to 10 c.c. of the urine add 50 c.c. of Fehling's solution, and treat as for sugar in food.

*Blood.*—Pour about two inches of urine in a test-tube, and add KOH solution. The precipitated phosphates, which in normal urine are white, are colored red or a rose tint if blood is present.

*Bile.*—A urine that retains its foam for a long time after shaking indicates the presence of bile. Such urines are usually yellow in color.

If fuming, nitric acid is added to urine in a test-tube in such a way that it shall flow down through the urine, the formation of a green ring at the junction of the two fluids indicates bile, and below this ring may appear blue, violet, and yellow rings.

*Questions.*—What reaction takes place in determining the urea? What is the gas measured in the urinometer? In what form is the uric acid in the urine? Why is HCl added before standing aside to crystallize? What is indican? What reaction takes place on adding the calcium hypophosphite? In what way does the chloroform act? Why avoid an excess of the hypophosphite? What is the difference between the alkaline and the earthy phosphates? Why, on the addition of the alkali, are not the earthy phosphates precipitated?



# APPENDIX.

## TABLE OF ELEMENTS.

ATOMIC WEIGHTS BASED ON CLARKE'S RECALCULATION, 1897.

Elements.	Symbols.	Valence.	Atomic Weight.		When Discovered.	By Whom Discovered.
			H = 1	O = 16		
Aluminum .....	Al	III	26.91	27.11	1827	Wöhler
Antimony .....	Sb	III-V	119.52	120.43		
Argon .....	A	....	?	?	1894	Rayleigh & Ramsey
Arsenic .....	As	III-V	74.44	75.01	1694	Schröder
Barium .....	Ba	II	136.39	137.43	1808	Davy
Bismuth .....	Bi	III-V	206.54	208.11		
Boron .....	B	III-V	10.86	10.95	1808	Lussac & Thénard
Bromin .....	Br	I-VII	79.34	79.95	1826	Balard
Cadmium .....	Cd	II	111.10	111.95	1817	Stromeyer
Calcium .....	Ca	II	39.76	40.07	1808	Davy
Carbon .....	C	IV-II	11.91	12.00		
Cerium .....	Ce	IV	138.30	139.35	1826	Mosander
Cesium .....	Cs	I	131.89	132.89	1860	Bunsen
Chlorin .....	Cl	I-VII	35.18	35.45	1774	Scheele
Chromium .....	Cr	II-III-VI	51.74	52.14	1797	Vauquelin
Cobalt .....	Co	II-VI	58.55	58.99	1735	Brandt
Columbium .....	Cb	III-V	93.02	93.73	1801	Hatchett
Copper .....	Cu	II	63.12	63.60		
Erbium .....	E	III	165.06	166.32		
Fluorin .....	F	I	18.91	19.06	1886	Moissan
Gadolinium .....	Gd	...	155.57	156.76		
Gallium .....	Ga	II-IV	69.38	69.91	1875	Boisbaudran
Germanium .....	Ge	IV	71.93	72.48	1886	Winkler
Glucinum .....	Gl	....	9.01	9.08	1828	Wöhler
Gold .....	Au	III	195.74	197.23		
Helium .....	He	...	?	?	1895	Ramsey
Hydrogen .....	H	I	1.00	1.008	1766	Cavendish
Indium .....	In	III	112.99	113.85	1863	Reich & Richter
Iodin .....	I	I-VII	125.89	126.85	1811	Courtois
Iridium .....	Ir	II-IV-VI	191.66	193.12	1804	Tennant
Iron .....	Fe	II-III-VI	55.60	56.02		
Lanthanum .....	La	IV	137.59	138.64	1839	Mosander
Lead .....	Pb	II-IV	205.36	206.92		
Lithium .....	Li	I	6.97	7.03	1817	Arfvedson
Magnesium .....	Mg	II	24.10	24.28	1808	Davy
Manganese .....	Mn	II-VII	54.57	54.99	1774	Gahn
Mercury .....	Hg	II	198.49	200.00		
Molybdenum .....	Mo	VI	95.26	95.99	1782	Hjebu

Elements.	Symbols.	Valence.	Atomic Weight.		When Discovered.	By Whom Discovered.
			H = 1	O = 16		
Neodymium.....	Nd	....	139.70	140.80		Welsbach
Nickel.....	Ni	II	58.24	58.69	1751	Cronstedt
Nitrogen.....	N	III-V	13.93	14.04	1772	Rutherford
Osmium.....	Os	IV-VI	189.55	190.99	1804	Tenonant
Oxygen.....	O	II	15.88	16.00	1774	Priestley
Palladium.....	Pd	II-IV-VI	105.56	106.36	1803	Wollaston
Phosphorus.....	P	III-V	30.79	31.02	1669	Brandt
Platinum.....	Pt	II-IV-VI	193.41	194.89	1750	Watson
Potassium.....	K	I	38.82	39.11	1807	Davy
Praseodymium...	Pr	....	142.50	143.60		Welsbach (?)
Rhodium.....	Rh	II-IV-VI	102.23	103.01	1803	Wollaston
Rubidium.....	Rb	I	84.78	85.43	1800	Bunsen
Ruthenium.....	Ru	IV-VIII	100.91	101.68	1845	Claus
Samarium.....	Sm	...	149.13	150.26		
Scandium.....	Sc	IV	43.78	44.12	1879	Nilson
Selenium.....	Se	II-IV-VI	78.42	79.02	1817	Berzelius
Silicon.....	Si	IV	28.18	28.40	1822	"
Silver.....	Ag	I	107.11	107.92		
Sodium.....	Na	I	22.88	23.05	1807	Davy
Strontium.....	Sr	II	86.95	87.61	1808	"
Sulfur.....	S	II-IV-VI	31.83	32.07		
Tantalum.....	Ta	V	181.45	182.84	1802	Eckeburg
Tellurium.....	Te	II-IV-VI	126.52	127.49	1798	Klaproth
Terbium.....	Tr	....	156.80	160.00		
Thallium.....	Tl	I-III	202.61	204.15	1861	Crookes
Thorium.....	Th	IV	230.87	232.63	1828	Berzelius
Thulium.....	Tm	...	169.40	170.70		
Tin.....	Sn	IV	118.15	119.05		
Titanium.....	Ti	IV	47.79	48.15	1824	Berzelius
Tungsten.....	W	IV	183.43	184.83	1785	d'Elhujar
Uranium.....	U	VI	237.77	239.59	1840	Pelagot
Vanadium.....	V	V	50.99	51.38	1867	Roscoe
Ytterbium.....	Yb	IV	171.88	173.19		
Yttrium.....	Y	III	88.35	89.02		Berzelius
Zinc.....	Zn	II	64.91	65.41		Valentine
Zirconium.....	Zr	IV	89.72	90.40	1825	Berzelius

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